SYNTHESIS AND CHARACTERIZATION OF A SMART SUPER ABSORBENT HYDROGEL

A THESIS SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENT FOR THE DEGREE OF **BACHELOR OF TECHNOLOGY**

IN

BIOTECHNOLOGY

Submitted by

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DECLARATION

I hereby state to the best of my knowledge and belief that the project entitled "**SYNTHESIS AND CHARACTERISATION OF A SMART SUPER ABSORBENT HYDROGEL**" is a record of original work carried out by me under the supervision of **Prof. S.S. Ray**, Dept. of Biotechnology & Medical Engineering, NIT, Rourkela. The results embodied in this project report are solely for academic purpose.

Date:

Place:

Smaranika Panda

ACKNOWLEDGEMENT

In the presentation of this report I recall with a sincere gratitude each of those who have been a source of immense help and inspiration during the process of my project work.

I take this opportunity to express my profound gratitude and deep regards to my supervisor **Dr. S.S. Ray** for his exemplary guidance, monitoring and constant encouragement throughout the course of this project. The blessing, help and guidance given by him time to time shall carry me a long way in my academic career.

I am indebted to all faculty members of the Dept. of Biotechnology and Medical Engineering for their advice and cooperation during the course of my study. I would like to express my sincere thanks to **Dr. Kunal Pal** for their constant help and for allowing me to use their instruments and materials. I am also thankful to other departments for permitting access to use their instruments.

Last but not least, I thank the almighty, my parents, seniors and lab mates for their constant encouragement without which this project work would not be possible.

Smaranika Panda

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CERTIFICATE

This is to certify that the thesis entitled **"SYNTHESIS AND CHARACTERISATION OF A SMART SUPER ABSORBENT HYDROGEL"** submitted by Ms. SMARANIKA PANDA in partial fulfillment of the requirements for the award of the degree of Bachelor of Technology in Biotechnology at the National Institute of Technology, Rourkela is an authentic work carried out by her under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/ Institute for the award of any Degree or Diploma.

Date:

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CONTENTS

LIST OF ABBREVIATIONS

%A	Percent Absorbance
Vol %	volume %
DMEM	Dulbecco's Modified Eagle medium
MEM	Minimal Essential Medium
kV	Kilo Volt
mA	Mili Ampere
MTT	3-(4,5-Dimethylthiazol-2-yl)-2,5-Diphenyltetrazolium Bro
M	Molar
nm	nanometer
µg	microgram
ml	mililitres
µl	microlitre
h	Hour
Sec	Seconds
θ	Theta

TEMED	N,N,N,N-tetramethylethelenediamine
APS	Ammonium Per Sulfate
MBAA	N,N-methylenebispolyacrylamide

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ABSTRACT

This research work aims to give an overview of the synthesis of a smart super absorbent semi interpenetrating hydrogel and their characterizations. The main objective is to mix polyacrylamide with Tamarind gum and to check how the parameters are varying to create a good strength sustained delivery system. We tried to optimize the process parameters of polyacrylamide and tamarind gum semi interpenetrating hydrogel. Swelling measurements were done and mechanical properties were investigated. Cyclic compression, creep & stress relaxation properties are observed. XRD and FTIR studies were analyzed, Biocompatibility is checked and cross linking density is calculated. Tamarind gum converted the polyacrylamide into a super absorbent. With the increasing concentration of tamarind gum, cross-link density increases. Addition of Tamarind gum increased the swelling capability of polyacrylamide gel and increased its pH sensitivity. From the mechanical test data, we can conclude that with increasing concentration of tamarind gum viscoelastic property is increased. From the XRD pattern, it is seen that the composite is highly amorphous. From the FTIR analysis, we can say that all the components used for making the composite are present. With the increasing concentration of tamarind gum, biocompatibility of the hydrogel increases.

Keywords: Superabsorbent, smart hydrogel, semi-interpenetrating, tamarind gum, polyacrylamide

Chapter 1

INTRODUCTION

1.1 Introduction

Biocompatible and biodegradable materials are effectively utilized as a part of tissue engineering for replacement of connective tissue and used for drug delivery. Polysaccharide based hydrogels has been widely studied for tissue engineering applications. This is because of the ease, simple accessibility, non-toxic and biodegradable nature of the polysaccharides. Gum tamarind is a galactoxyloglucan isolated from seed kernel of Tamarindus indica [1]. Tamarind gum has numerous particular properties and has been applied in various fields.

1.2 Properties of tamarind gum

Properties of tamarind gum are biocompatible, antibacterial, high viscosity, high PH tolerance, adhesive, and so on [2]. Due to these properties, it is extensively used in thickening, stabilizing and jelling agent in the food and pharmaceutical industries [3].

In addition to these, there are many other important properties of Gum Tamarind. They are non carcinogenic, mucoadhesive, can hold high drug and are highly thermal stable [4]. This led to its application in localized drug delivery system.

But the drug carrying capability of the above polymers is less, which is not a good property for the sustained drug delivery system. Also, the mechanical strength of the polymeric film is less. So to improve these two properties, we have prepared a semi IPN (interpenetrating) of this with polyacrylamide.

1.3 Why polyacrylamide?

Polyacrylamide has already its applications in contact lenses, sustained released wound dressing material [5]. Polyacrylamide and its derivatives are well known for their hydrophilic & inert nature. Also, it provides inherent protection to proteins to be used in various applications, like Glucose sensors. It is highly water absorbent. It is also used as a thickener and suspending agent [5].

1.4 Theory behind hydrogels

The most common definition of hydrogel is, it is a cross-linked polymeric network of one or more monomers that are hydrophilic [6]. They have the ability to swell in water and retain some amount of water within it but do not dissolve in water. Due to their large water content they are very flexible like in natural tissue Hydrogels have a wide range of applications. They are used for tissue engineering, drug delivery, wound care products, elements of implants, etc.

An IPN is a polymer comprising two or more networks which are at least partially interspersed or crossed intricately together on a polymer scale but not covalently bounded to each other by any chemical bond. In a semi interpenetrating gel, one is getting cross-linked but the other not. In our gel, polyacrylamide is cross-linked but not tamarind gum.

A hydrogel is said to be superabsorbent if they can absorb and retain extremely large amounts of a liquid relative to their dry volume. They can absorb water up to thousand times of their dry volume while maintaining the three dimensional network structures. Due to the hydrophilic functional groups attached to the polymeric backbone, they swell. They are resistant to dissolution due to the cross-links between net-work chains.

In hydrogel, the polyacrylamide chains form a network by covalent cross-links.

1.5 Application of smart hydrogel

Smart hydrogels are used in drug delivery systems, injectable hydrogels, tissue engineering, actuators, sensors and self- healing.

1.6 Application of superabsorbents

Superabsorbents can be used in diapers and sanitation napkins, surgical pads, wound dressings. It can be also used in agriculture because it can retain water for supplying it to plants. In industry, it can be used to dispose liquid waste.

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Chapter 2

REVIEW OF LITERATURE

LITERATURE REVIEW:

Sl NO	First author etal, year, Journal	Composition (conc.)	Process (cross linker, conc.)	Characterization Techniques	Outcome
1	Fei Dong etal, 2012, Journal of material chemistry	Hyalliosite, AA, acrylamide	Inverse suspending process	SEM, TEM, FTIR, XRD, BET, TG	humidity control
2	Sajesh K. M. etal, 2013, International Journal of Biological Macromolecules	ppy-Alg, chitosan, fecl3(O.A.)	By using alginate blend and chitosan, a scaffold is prepared. This is used for bone tissue engineering	SEM, FTIR, SECM, Alamar blue assay	Used in bone tissue engineering
3	J.L.Shamshina etal, 2014, Journal of material chemistry B	chitin, calcium alginate, alginic acid	Calcium alginate and chitin fibres were prepared by dry-jet wet spinning method.	SEM, TEM, FT-IR	The composite can be used for wound dressing.
4	Sanil Sebastian etal, 2014, Journal of water resource and protection.	sodium alginate layered anionic clay (MgAl)	Adsorption properties of layered anionic clay composites for the removal of anionic dyes from water were studied.	XRD, SEM, Autosorb 1c from Quantachrome instruments	water/waste water treatment

5	Niyaz Mohammad Mahmoodi, 2013, Journal of the Taiwan Institute of Chemical Engineers	Magnetic ferrite nanoparticle-alginate	Synthesis of MFN-alginate, Characterisation& Dye absorption	XRD, SEM, FT-IR	Synthesis, characterization & binary system dye renoval.
6	Han Y etal, 2013, Journal of ActaBiomater	calcium silicate-alginate	A hydrogel is prepared by using sodium alginate and calcium silicate.	SEM, FT-IR	It can be used as injectable hydrogels
7	Ibrahion M.EI Sherbiny etal,2013, ELSEVIER	New calcareous soil-alginate composites	collection of the brown macro-algae biomass, characterisation of extracted alginates from algae, ch.Ca.soil, preparation of alginate hydrogel particles, preparation of cal. Soil-alginate	FT-IR, UV- VIS, EA	It can easily uptake Iron, Mangenese and arsenic from water.
8	T.Coradinetal, 2013, Appiled Microbiology and Biotechnology	silica-alginate	coacervation and layer-by-layer process	NA	Application of micro-encapsulation to bioartificial organ elaboration
9	Mariana Lonitaetal, 2013, carbohydrate polymers	Sodium alginate/Graphene oxide(AI/GO)	Sodium alginate and grapheme oxide film was prepared by Casting.	FT-IR, XRD, SEM ,TEM, TG	The film's structure, mechanical properties, thermal properties, etc were studied.

10	ChihHui Yang etal, 2014, Molecules	Magnetic Pycnoporussanguineus-Loaded Alginate	Alginate beads were used to remove a dye and it is loaded with magnetic Pycnoporussanguineus.	NA	Removing dye from aqueous solution
11	MdShahidul Islam, 2012, Dhaka University Journal of Science	Nanofibers of the composite of alginate (Alg) and carbon nanotube (CNT)	Electrospinning method	SEM, TEM,mechanical measurement, Electrical conductivity	Fabrication of biomaterial scaffolds.
12	Sa-Ad Riyajanetal, 2013,Journal of Material Science	A composite of Sodium alginate or natural rubber and coconut waste	The composite is used to remove lead from waste water. This was prepared by using Sodium alginate or natural rubber and coconut waste.	ATR-FTIR, and SEM	Adsorption of pb(II)ions
13	Deu Gao,1999, Journal of material chemistry	Polyacrylamide- Bentonite composite.	Dynamic and transient rheological properties were measured for a series of hydrogel composites whose microstructure has been reported previously.	NA	Rheological properties of poly(acrylamide)-bentonite composite hydrogels

14	Ashwani Goyal etal, 2014, British Journal of pharmaceutical research	Acrylamide-starch silicon dioxide	For preparing the porous hydrogel,Gas blowing method was used.	NA	A superporous hydrogel is synthesized.
15	Bhavesh D. Kevadiya etal, 2013, Biochemistry and Biophysics (BAB) Volume 1 Issue 3,	polyacrylamide/Na+-MMT (Montmorillonite)	By free radical polymerization, the hydrogel is developed. AM and MMT are added in the ratio of 6:1, APS,10% and TEMED were added	XRD, FTIR, NMR, TGA	Can be used in anticancer drug carrier
16	Priyanka Goyaletal, 2015, Designed Monomers and Polymers	polyhydroxyethyl methacrylate (pHEMA) and methyl cellulose (MC)	0.45g of HEMA and 1g of MC, 0.025ml of 20% APS and 0.025ml of TEMED	XRD, FTIR, swelling, mechanical and electrical studies	Hydrogels are biocompatible, Viscoelastic, has resistive dominat behaviour and moisture retention capacity
17	Shriram S Rohokale1 etal, 2012, Journal of Chronotherapy and Drug Delivery	Polysaccharide hydrogels from the seeds of Tamarindus indica and from the trunk of Prunus amygdalus	For different phytochemicals, purity is tested by doing chemical tests for the two hydrogels.	FTIR, SEM	Hydrogels can be used as excipients.

18	Peng Li etal, 2009, Composites: Part B,	Polyacrylamide, laponite clay	Hydrogels were synthesized using a monomer (AAm), clay is used as a cross-linker, APS and TEMED as accelerator.	FTIR, XRD, Swelling kinetics	Swelling studies are done and the gel is found to be pH sensitive. Swelling capacity was increased with temperature.
19	Erdener Karadag etal ,2002, Turk J Chem	Acrylamide,Sodium Acrylate	0.1 g in 1 mL of distilled water, 0.2 mL of APS, 0.25 ml of TEMED	Swelling studies	The hydrogel can be used as a super water retainer and can be used in various fields like agriculture and industries.

Table 1. Literature review

Chapter 3

OBJECTIVES AND WORK PLAN

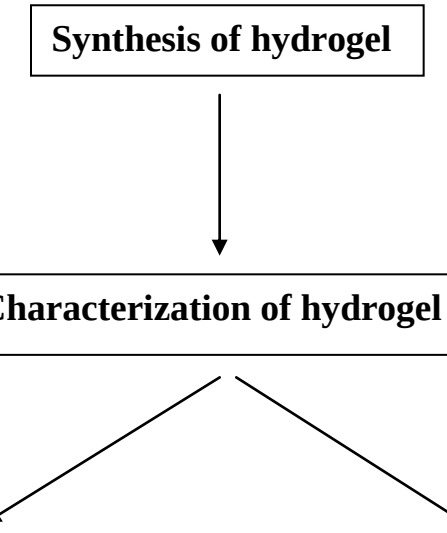
3.1 Aim

To synthesize and characterize a smart superabsorbent semi-interpenetrating hydrogel using tamarind gum and polyacrylamide.

3.2 Objective

- To optimize the process parameters of polyacrylamide and tamarind gum semi interpenetrating hydrogel.
- To characterize the prepared hydrogel.

3.3 Work plan



Physiochemical	Biological
Swelling study	Biocompatibility
Mechanical behavior	
FTIR	
XRD	
Cross-link density	

Chapter 4

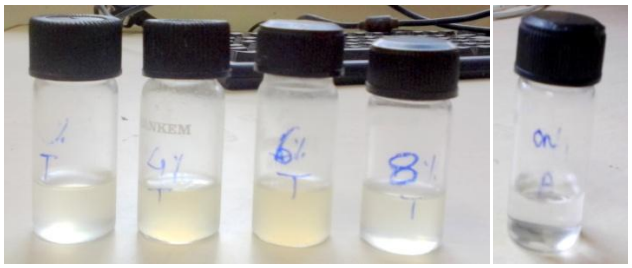
MATERIALS AND METHODS

4.1 Materials

Tamarind gum powder was procured from Maruti Hydrocolloids, India. Polyacrylamide and APS were procured from Rankem, Delhi, India. TEMED and MBAA are procured from Hi Media Laboratories Pvt Ltd, Mumbai, India. For the whole work, distilled water was used.

4.2 Preparation of tamarind gum-polyacrylamide hydrogel

2%, 4%, 6% & 8% tamarind gum was prepared. 20% polyacrylamide was prepared. Tamarind gum and polyacrylamides were mixed in the ratio of 4:1. APS is added at 0.0012 that of polyacrylamide as a photo initiator for polyacrylamide. The weight of the covalent cross-linker, MBAA, was taken as 0.0006 that of polyacrylamide [7]. N,N,N',N'- tetramethylethylenediamine is added at 0.0025 the weight of polyacrylamide [7] , as the cross linking accelerator for polyacrylamide. These are placed at room temperature for 24 hours. Polyacrylamide was taken as control. The same procedure was repeated.



(a) (b) (c) (d) (e)

Fig. 1. Pictures of the prepared hydrogels: (a) 2% (b) 4% (c) 6% (d) 8% (e) control

4.3 Swelling studies

The samples are placed in vacuum dried for 24 hours. Weight of the dry hydrogels is taken using a weighing balance & then immersed in distilled water at room temperature. They are taken out at pre-defined time intervals, excess water was removed using filter & then final weight is measured by a sensitive weighing balance. The degree of water sorption is calculated in terms of the swelling % as calculated below:-

Swelling %= $(W_s-W_d) / W_d$

Where, W_s=Weight of swollen hydrogel and W_d= Weight of dry hydrogel.

The swelling % with respect to different time intervals is plotted with swelling ratio in Y-axis & time interval in X-axis.

Buffer of pH 1.92(2g of NaCl was added to 1000ml distilled water & then 7ml of Hcl is added) & pH 9.5(1M Tris buffer was prepared) were prepared and swelling studies are calibrated as above.

4.4 Cross-Link Density

Hydrogel samples were prepared according to the above procedure in cylindrical shapes. The hydrogels were kept at room temperature for 24 hours for cross-linking. Using the Flory-Rehner equation, the average polymer molecular weight in between two cross-links were calculated [8,9]:-

$$-[\ln\ 1-v_{2,s}\ +v_{2,s}+\ \chi_1 v_{2,s}^2]\ =\ \frac{V}{vM_c}\ \ 1-\frac{2M_c}{M_n}\ \ \ v_{2,s}^{\frac{1}{3}}-\frac{v_{2,s}}{2}$$

Where $M_n$is the molecular weight of the polymer before cross-linking, M_c is the average molecular weight of polymer between two cross-links, v is the specific volumeof the hydrogel before swelling, V is the molar volume of the solvent (water,18 ml/mol), $v_{2,s}$is the volume fraction of the polymer in the swollen state and calculated by the inverse of Volume swelling ratio, χ_1 is the Flory–Hugginsparameter describing the polymer–solvent interaction [10]. The crosslink density q was calculated using M_c [11]:-

$$q = \frac{M_n}{M_c}$$

The parameter $v_{2,s}$ was determined from the volume-swelling ratio q_v [11]:

$$v_{2,s} = \frac{1}{q_v}$$

The volume-swelling ratio was calculated as [11]:

$$q_v = 1 + \frac{q_w - 1 \times \rho_2}{\rho_1}$$

where ρ_2 and ρ_1 are the densities of the polymer network and solvent, respectively.

The weight-swelling ratio q_w was determined from [11]:

$$q_w = \frac{m_s}{m_0}$$

4.5 Mechanical Tests

The samples are poured into a plastic mould (cylindrical of height 3cm & area 144.7cm²). The stress-relaxation, compression, creeps were evaluated using Stable Micro system Texture analyzer.

4.5.1 Stress-relaxation

Under a constant strain how an applied stress is relaxed is observed. The graph of normalized force vs. time was plotted. The data of stress relaxation was fitted using the following equation.

P (t) = P0+P1 exp(-t/τ1)+P2exp(-t/τ2)+P3exp(-t/τ3) **(Pelegs Eqn)** [12]

Where P (t) represents the magnitude of decaying parameter. It may be force or stress or modulus at time t, P0 is the magnitude of the residual stress after t =1, and Pi and si are the i-th term constants the function.

4.5.2 Creep

Creep is an important phenomena observed in viscoelastic materials. The distance vs. time is plotted and the using Maxwell's eqn. [13], the % recovery is calculated.

$$J(t) = J_0 + J_1\left[1 - \exp\left(-\frac{t}{T_{ret}}\right)\right] + \frac{t}{\eta_0}$$

(Maxwell's equation)

4.5.3 Compression

The cyclic compression is done for 10 numbers of cycles at 1mm/sec. The peak force and resilience is calculated from the data obtained from Exponent lite. [14]

4.6 XRD studies

The hydrogels were prepared and dried in a vacuum drier at 37ºC. The samples were taken in powder form. XRD analysis were done by XRD (Rigaku ultima IV) machine in 2θ range of 5-35^o at 5º/min in Bragg Benton mode. The source of the X-ray was Cu-Kα. The operating voltage was 40Kv and current was 40mA.

4.7 FTIR studies

By FTIR studies, the interactions between the functional groups of the polyacrylamide and gum tamarind were analyzed. Hydrogel pellets were used for the FTIR. FTIR analysis were done by FTIR spectrometer (Alpha-E, Bruker, Germany), attached with a ZnSe ATR cell. In the wave number range of 4000cm^{-1} – 400cm^{-1}, the samples were analyzed. The original spectra were smoothened.

4.8 Biocompatibility

Fresh samples were prepared. 100µl of the sample is added in 96 well plates in aseptic manner. It is placed in UV-radiation for 20 minutes for sterilization followed by treatment with 70% ethanol for 20 minutes to remove any contaminants. The samples were washed thrice with PBS to remove ethanol and any un reacted monomer. The samples were washed with incomplete media (DMEM, low glucose).

Adipose derived stem cells are used for assessing the biocompatibility. ADSCs were maintained in low glucose DMEM media supplemented with 10% FBS and 1% antibiotics. At confluency the cells were trypsinized and 10^4 cells/well were seeded. The ADSCs were cultured in complete media (DMEM, low glucose, supplemented with FBS) under 5% CO_2 atmospheric conditions. Samples were assessed for their bio-compatibility for 48 hours. The presence of viable cells was determined through MTT [15].

Chapter 5

RESULTS AND DISCUSSIONS

5.1 Superabsorbency of the hydrogel

The dry hydrogel has swollen so much.

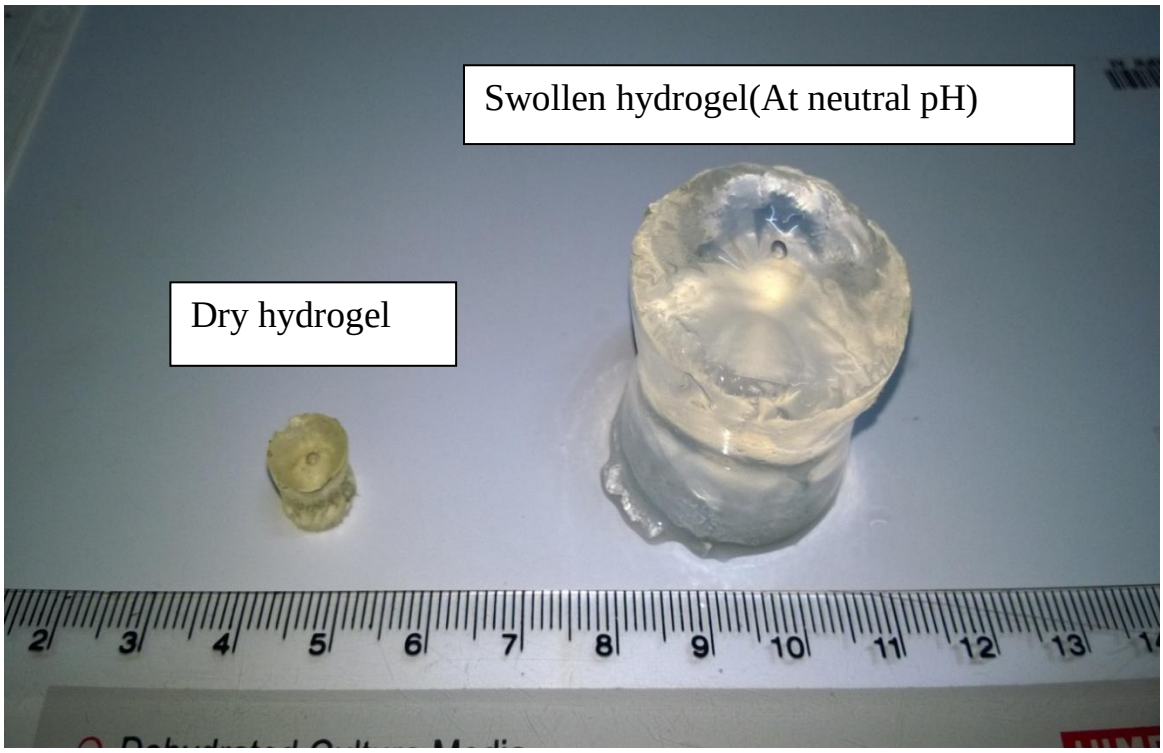


Fig. 2. Hydrogel as a superabsorbent

Volume of dry hydrgel:-

r= 3mm, h=8mm

Volume= 226.08mm³

Volume of Swollen hydrogel:-

r= 12.5mm, h= 25mm

Volume= 12265.625mm³

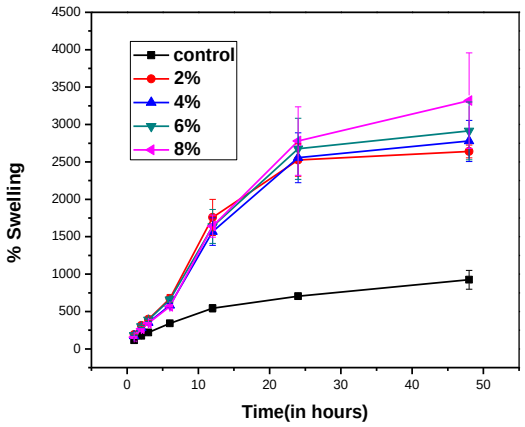
The swollen ratio= 12265.625/226.08= 54.25±8

The gel absorbs 54.25 times its own volume. So, it can be considered as a superabsorbent

5.2 Swelling kinetics

Swelling test is done to know how much water a hydrogel can absorb?

5.2.1 Swelling test at pH 7



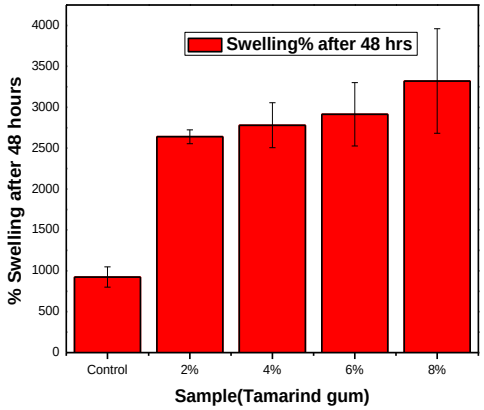
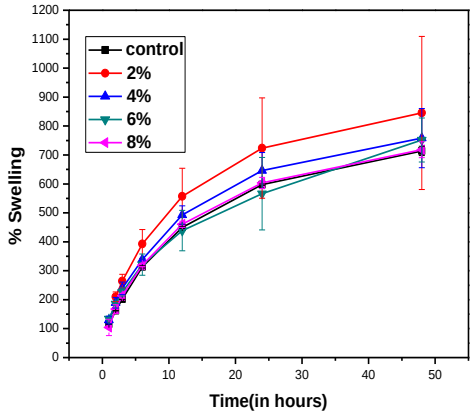


Fig. 3. % swelling vs. time at pH 7

Fig. 4. % swelling vs. sample at pH 7

All are swollen more than the control. So we can conclude that by adding tamarind gum, swelling capability of polyacrylamide hydrogel increases. It may be due to the presence of hydrophilic groups present in Tamarind gum. All are following a pattern. Control < 2% < 4% < 6% < 8%.

5.2.2 Swelling test at Acidic pH (1.3)



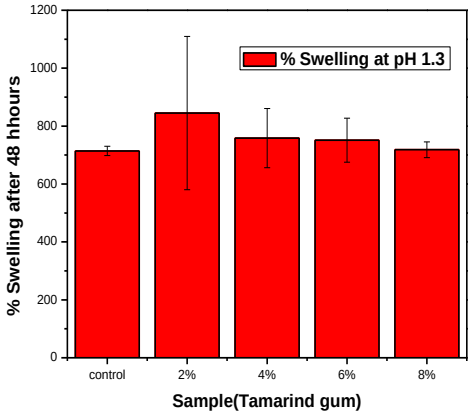


Fig. 5. % swelling vs. time at pH 1.3

Fig. 6. % swelling vs. sample

It is observed that all the sample de swells. We can say that addition of Tamarind gum converted the gel into more acidic pH sensitive gel [16]. All have swollen more than control. Further addition of Tamarind gum decreases swelling. This may be due to the cause that at a particular charge distribution, cross-linking density becomes more prominent. It is expected that cross-link density affects the mechanical behavior of hydrogels directly [17]. Hydrogels having high cross-link density have various channels to diffuse water but as cross-link density increases, water content decreases. From the cross-link data it is observed that with the addition of tamarind gum, cross-link density increases. So, their water holding capability decreases and hence swelling capability decreases.

5.2.3 Swelling test at Basic pH (9.3)

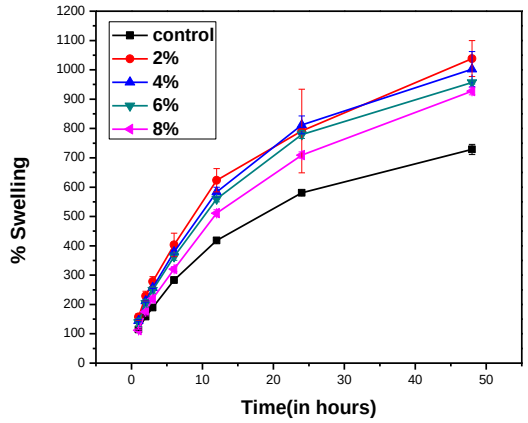


Fig. 7. % swelling vs. time at pH 9.3

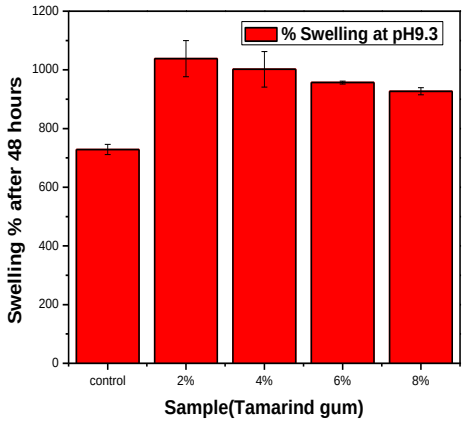


Fig. 8. % swelling vs. sample

Polyacrylamide gel swells but not that much of neutral pH. All are swelling more than control. Further addition of gum tamarind decreases swelling. The same reason as in the previous case holds true here, i.e., at a particular charge distribution the cross-link density becomes more prominent.

5.3 Cross-link density

The following key parameters are used for solving the equation:-

Parameter	Value of samples				
	2%	4%	6%	8%	Control
M (primary molecular mass)	71.08g/mol	71.08g/mol	71.08g/mol	71.08g/mol	71.08g/mol
v (Specific volume of hydrogel)	0.978 cm^3/g	0.909	0.883cm^3/g	0.818g/mol	0.808g/mol
V (molar volume of water)	18 cm^3/mol	18cm^3/mol	18cm^3/mol	18cm^3/mol	18cm^3/mol
$v_{2,s}$ (polymer volume fraction post-swelling)	experimental	experimental	experimental	experimental	experimental
ρ_2 (density of hydrogel)	1.021g/cm^3	1.1g/cm^3	1.133g/cm^3	1.221g/cm^3	1.236
ρ_1 (density of water)	0.998 g/cm^3	0.998g/cm^3	0.998g/cm^3	0.998g/cm^3	0.998g/cm^3
χ_1 (Flory solvent-polymer interaction term)	0.60 (approx.)	0.60(approx)	0.60(approx)	0.60(approx)	0.6(approx)

Table 2. Parameters used for determination of Cross-linking density

The average molecular weight of polymer after cross-linking (M_c) and the cross-linking density (q) is calculated and they are the following:-

Sample	**M_c**	**Cross-linking density (q)**
2%	39.23	1.81
4%	38.72	1.83
6%	38.4	1.85
8%	37.22	1.90
Control	36.19	1.96

Table 3. Cross-linking density of the hydrogels

It is observed that the cross-linking density of polyacrylamide is found to be more compared to other samples and with increasing concentration of gum tamarind the degree of polymerization increases.

5.4 Mechanical Tests

Mechanical tests are performed to know the elastic and inelastic behavior of the hydrogel when we apply a force on to it. By performing these tests, we can know whether a hydrogel can be suitable for its mechanical applications.

5.4.1 Stress relaxation

It describes how the hydrogel relax stress under constant strain. The non-linearity is described by SR studies. We can predict the firmness of a hydrogel from SR studies.

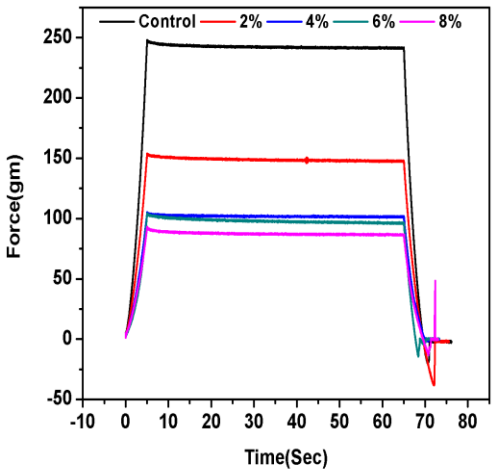


Fig. 9. Stress-Relaxation curve (force vs. Time)

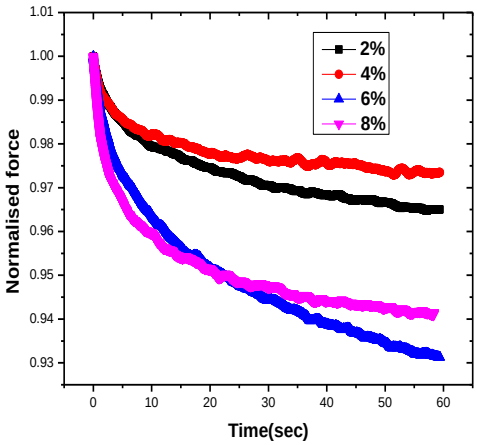


Fig. 10. Stress-Relaxation curve (normalized force vs. time)

Stress relaxation is more in case of 8%. Higher the concentration higher is the stress relaxation. This might be because of the increasing elasticity of the hydrogel. SR curve measures the viscoelastic nature of a material. When the stress relaxation is Zero then, the material is ideally elastic and if the stress relaxation is 100%, then the material is fluid in nature or ideally viscous. But our graph shows a stress relaxation in between them. So, we can say our hydrogel is viscoelastic in nature. We can conclude that the hydrogel is viscoelastic and with increasing concentration of tamarind gum the viscoelasticity increases.

Curve fitting using Peleg's equation

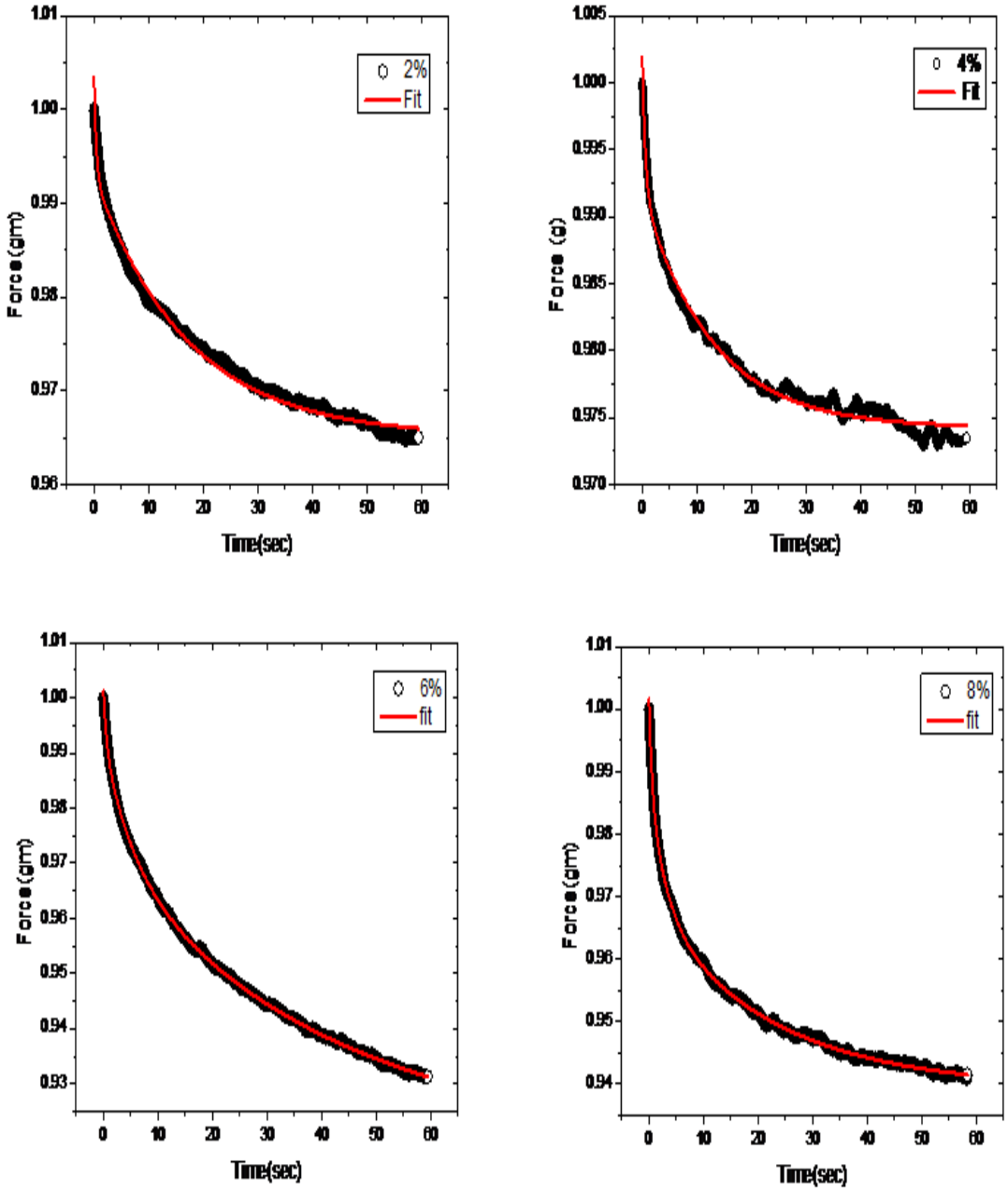


Fig. 11. Curve fitting to Peleg's equation

5.4.2 Creep

Creep describes the elastic behavior and non-linearity fashion of a material. Under a constant stress, how polymer strains is described.

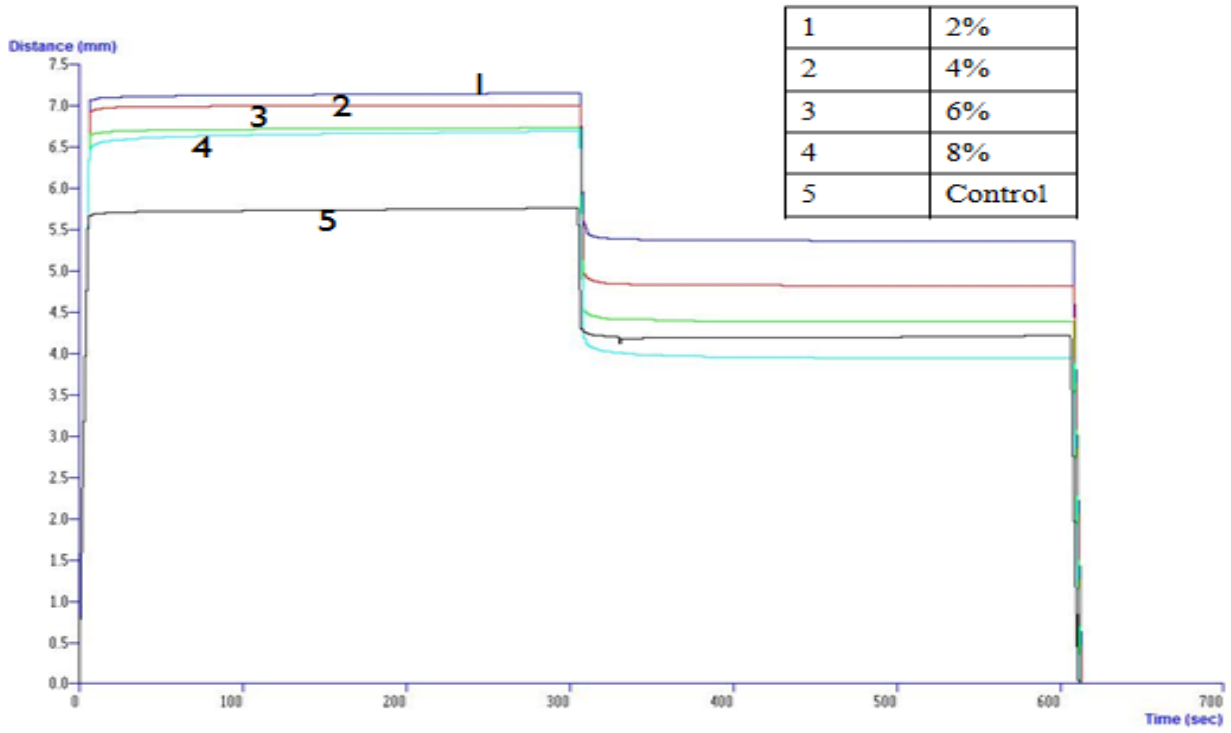


Fig. 12. Creep studies of hydrogels

The order is 2 %< 4 %< 6 %< control<8%. It is observed that with the increased concentration of Tamarind gum, the creep is increasing except the control. So, we can conclude the viscoelastic property of the hydrogel is increasing with the addition of tamarind gum. It is observed from the graph that the change in strain is more in case of 8%. There is some error in case of control but all other are following a pattern. The results indicated that there is an increase in elastic component of hydrogel with increased concentration of Tamarind gum.

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Sample	Maxwell's Equation	% Recovery
Control	J(t)=2.051484-(t/200)	75.85406
2% Tamarind gum	J(t)= 2.560981+(t/0.00011)	43.72554
4% Tamarind gum	J(t)= 2.512845+(t/9.14913E-05)	44.95151
6% Tamarind gum	J(t)= 2.408532+(t/0.000104)	51.12034
8% Tamarind gum	J(t)= 2.33799+(t/0.000215)	55.92056

Table 4. % Recovery of hydrogels in creep

From the creep studies, we can know how a material is able to recover immediately to its original shape when the stress is removed [18]. The % recovery provides information about the elastic component of the hydrogel. If a material is elastic, there will be 100% recovery. Our results have no 100% recovery. This may be due to the fact that the viscous phase is lost during the creep. It is observed that the order is 2% <4% < 6% <8% <control. We can conclude that the % recovery increases with the increased concentration of Tamarind gum. This might be due to the fact that as the elasticity is increasing the viscous property is decreasing.

5.4.3 Cyclic Compression

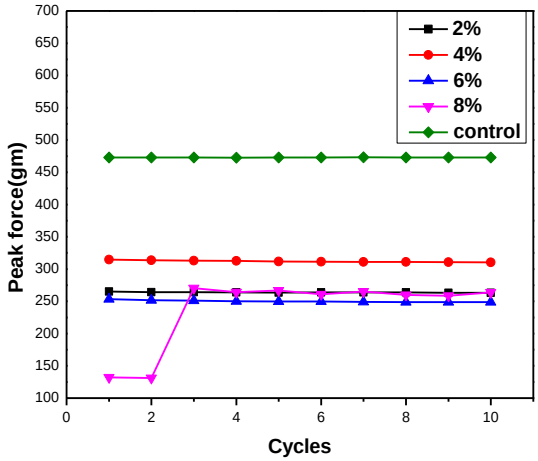

Fig. 13. Cyclic compression of hydrogels (Peak force vs. Cycles)

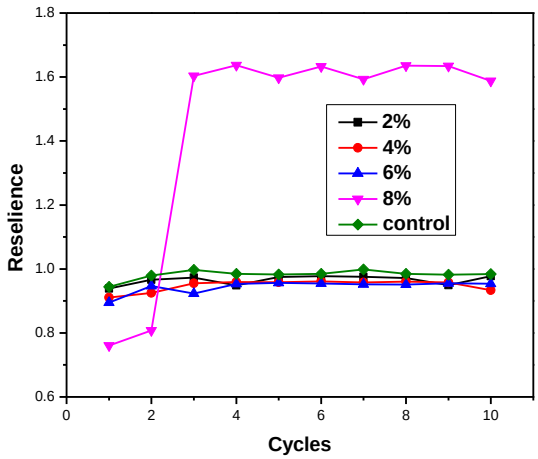

Fig. 14. Cyclic compression of hydrogels (Resilience vs. Cycles)

It is observed from the graph that force needed to compress decreases with addition of tamarind gum. There is an exception to 8%. This may be due to some experimental error. The peak force of control is highest. This might be due to the cross-link density factor. Cross-link density of control is found to be more. So its mechanical strength is more. Hence force needed to compress those increases.

5.5 XRD studies

XRD is used to know the arrangement of atoms or molecules in the internal lattice of crystalline structure.

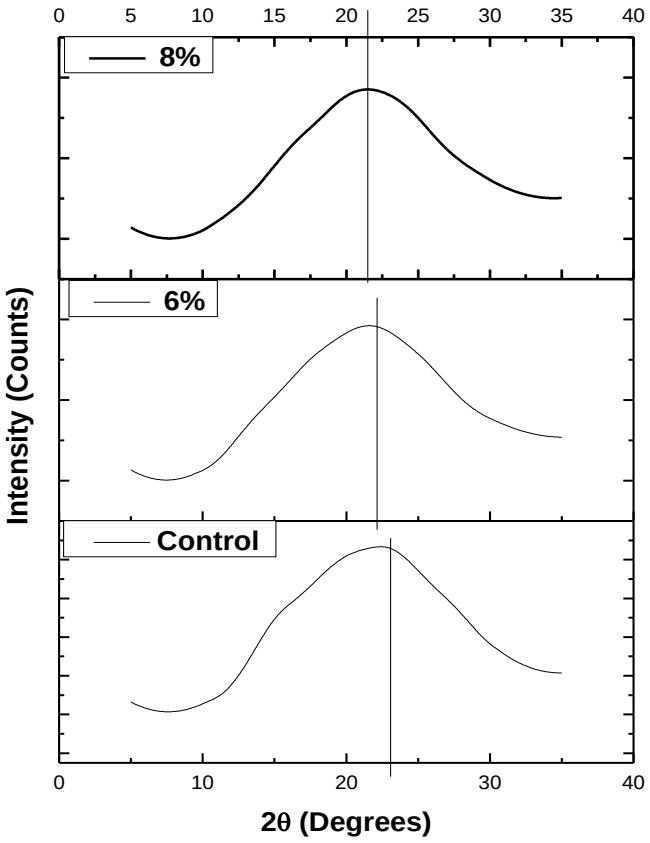

Fig. 15. XRD pattern of hydrogels

Broadening peak shows that the composite is amorphous. The peak nearer to 25º corresponds to the polyacrylamide [19, 20] and the peak at 20º corresponds to Gum Tamarind [21]. It is observed that with addition of gum tamarind, the peak shifts from 25º to 20º. So, we can conclude that all the components which were used to form the hydrogel are present.

5.6 FTIR Studies

FTIR study is used to know the presence of certain functional groups in a molecule and to determine the amount of components n a mixture.

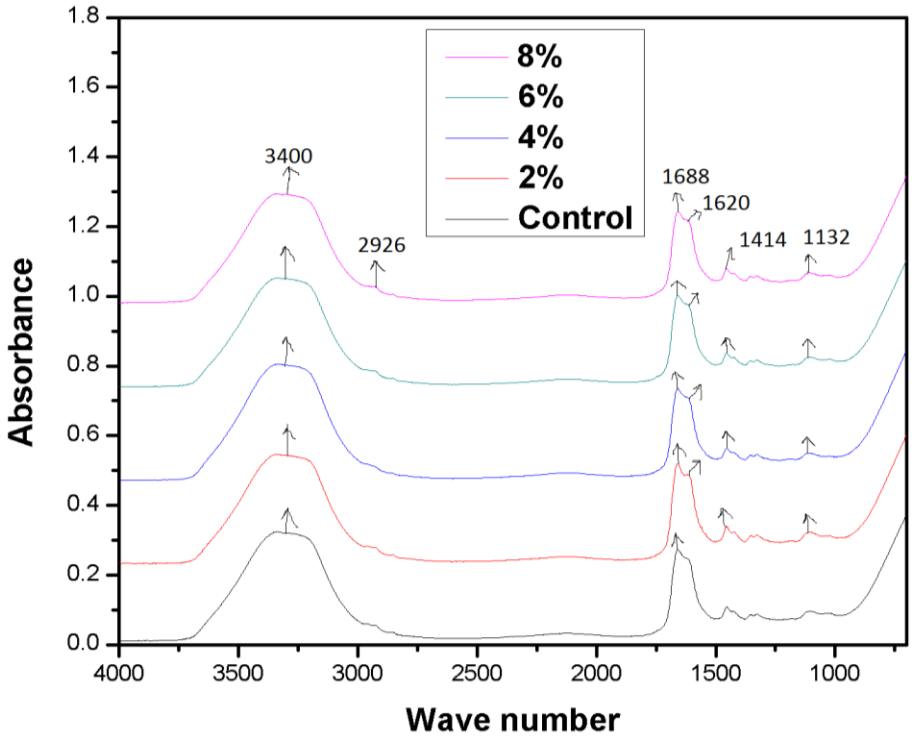

Fig. 16. FTIR spectra of hydrogels

The peak at 3400cm^{-1} corresponds to the -OH groups present in tamarind gum and polyacrylamide both [22]. Further, the peak around 1620 cm^{-1} and 1414cm^{-1} corresponds to asymmetric and symmetric stretches of COO^{-} for pure tamarind [23]. The peak at 1132cm^{-1} is due to C−O stretching [23]. The peak at 2926 cm^{-1} shows the presence of $–CH_2$ groups [23]. From the spectrum of polyacrylamide, we can say that a band at1688 cm^{-1} is attributed to the carbonyl (C=O) bond [24]. So, we can conclude that all the components which were used to form the hydrogel are present.

5.7 Biocompatibility

The biocompatibility study is used to know the viability of the cells. We can know whether the hydrogel is fit for human use and whether it has any harmful effects.

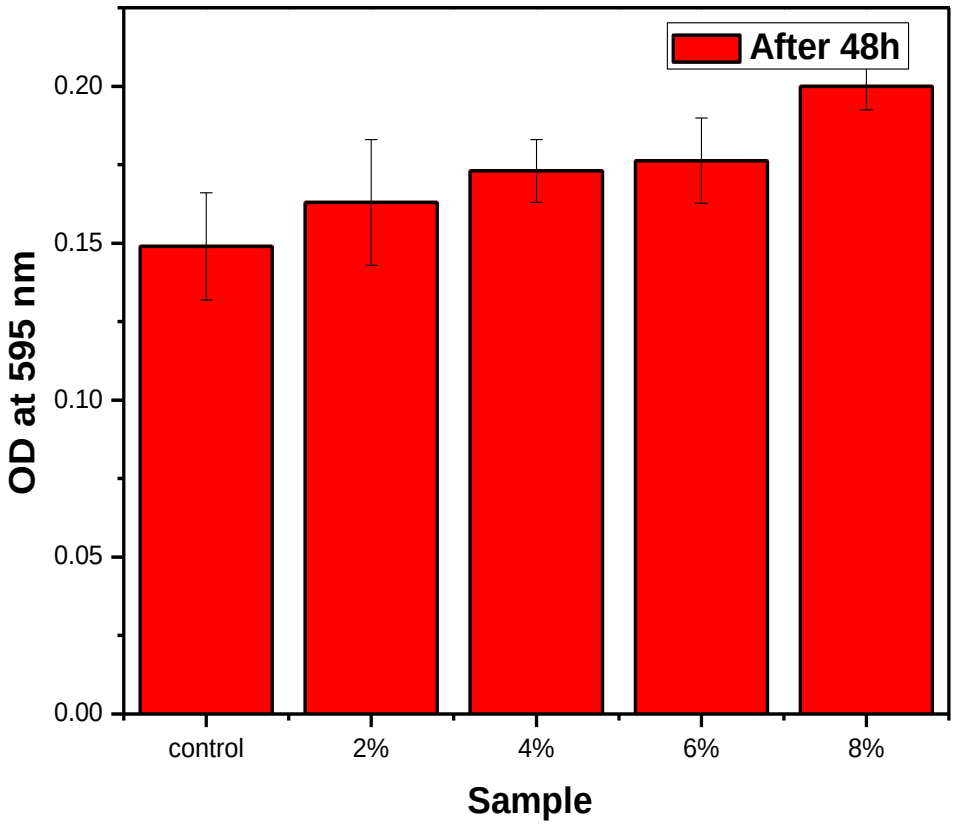

Fig. 17. Biocompatibility of hydrogels with ADSCs

It is observed that OD is increased in all the samples more than the control. It follows a pattern. Control<2%<4%<6%<8%. So, we can conclude that addition of Tamarind gum increases the cell viability.

5.8 Application (Gastric drug delivery system)

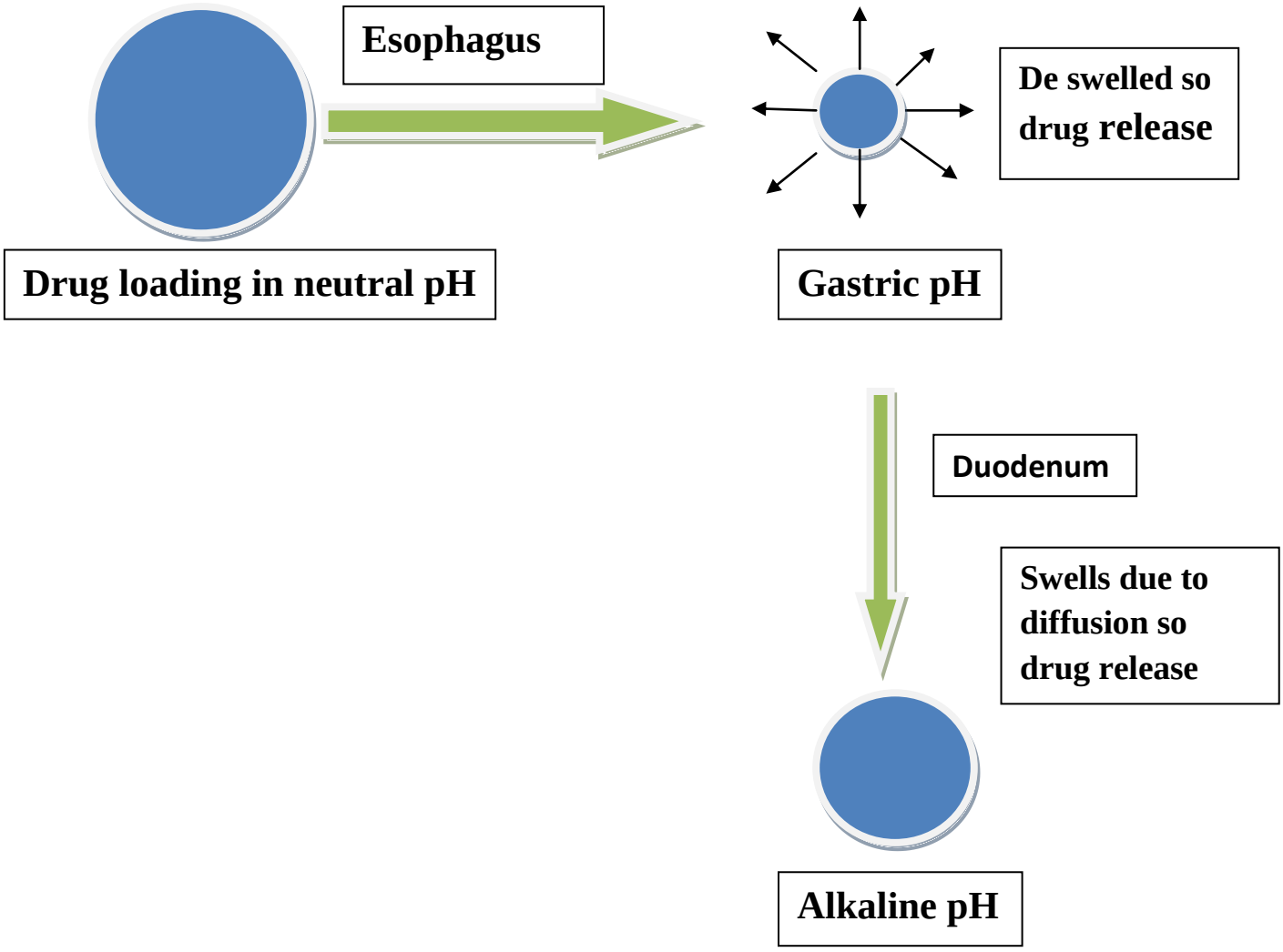

Fig. 18. Application of hydrogel in gastric drug delivery system

Chapter 6

CONCLUSION

Conclusion

Tamarind gum converted the polyacrylamide into a super absorbent. With increasing concentration of Tamarind gum, Cross-link density increases. Addition of Tamarind gum increases the swelling capability of polyacrylamide gel. In neutral pH, with increasing concentration of tamarind gum, the swelling capability increases but in acidic and basic pH, the swelling is more than polyacrylamide but with the increasing concentration of tamarind swelling decreases. This might be due to some charge distribution, cross-link density becomes more prominent. So, tamarind gum enhances the pH sensitivity of polyacrylamide which will be highly beneficial for gastric/duodenal drug delivery system. From the Mechanical test data, we can conclude that with increasing concentration of tamarind gum, viscoelastic property is increased. From the XRD pattern, it is seen that the composite is highly amorphous. From the FTIR analysis, we can say that all the components used for making the composite are present. With increasing concentration of tamarind gum, biocompatibility of the hydrogel increases. Swelling kinetics data shows that the polyacrylamide hydrogel de-swells in acidic medium and again swells in alkaline medium. So, if we will load the drug in neutral pH, when it will reach the stomach it will de-swell so drugs can be released and again in duodenum, it will re-swell and the residual drug remained in the hydrogel can be released. So, it can be used for Gastric drug delivery system. (Gastric or duodenal ulcer).

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